IS 8687-2 (1977): Methods of test for vitreous enamels and frits, Part 2: Fusion flow test [CHD 9: Ceramicware]

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IS : 8687 (Part 2) - 1977

Indian Standard

METHODS OF TEST FOR VITREOUS ENAMELS AND FRITS

PART I SIEVE ANALYSIS

UDC 666.293.5 : 620.168.32

INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

May 1978

AMENDMENT NO. 1 JUNE 2007
TO
IS 8687 (PART 1) : 1977 METHODS OF TEST FOR VITREOUS ENAMELS AND FRITS

PART 1 SIEVE ANALYSIS

(Page 3, *clause* **2.1,** *line* 1) — Substitute 'IS 2717 : 1979†' *for* 'IS 2717 - 1964†'.

(Page 3, footnote marked †) — Substitute the following for the existing:

'†Glossary of terms relating to vitreous enamelware and ceramic metal systems (*first revision)'*

(Page 4, *clause* **4.1.2,** *line* 1) — Substitute 'IS 460 (Part 1) : 1985*' *for* 'IS : 460 - 1962*'.

(Page 4, *footnote marked* *) — Substitute the following for the existing:

'*Specification for test sieves Part 1 Wire cloth test sieves *(third revision)'*

(Page 6, *clause* **5.1.2,** *line* 1) — Substitute 'IS 460 (Part 1) 1985*' *for* 'IS : 460 - 1962*'.

(*Page* 6, *footnote marked* *) — Substitute the following for the existing:

'*Specification for test sieves Part I Wire cloth test sieves (*third revision)*'

(CHD 9)

Reprography Unit, BIS, New Delhi, India

Indian Standard

METHODS OF TEST FOR VITREOUS ENAMELS AND FRITS

PART I SIEVE ANALYSIS

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Indian Standard

METHODS OF TEST FOR VITREOUS ENAMELS AND FRITS

PART I SIEVE ANALYSIS

0. FOREWORD

0.1 This Indian Standard (Part I) was adopted by the Indian Standards Institution on 10 October 1977, after the draft finalized by the Ceramicware Sectional Committee had been approved by the Chemical Division Council.

0.2 The fineness of frit in wet or dry-milled vitreous enamels or other ceramic coatings for metals has a direct bearing on many of its properties, such as fusibility, tearing, gloss, opacity, suspension in the slip and ease of spraying. Sieve analysis is the most convenient way of ascertaining fineness. Two methods of sieve analysis have been prescribed in this standard. The first (Method A) is the referee method which is meant for use where higher accuracy is required or for comparison of data obtained from different laboratories. The second (Method B) is the routine method which is meant for normal in-plant production control operations.

0.3 In the formulation of this standard assistance has been derived from Publication C-285-64 'Methods for sieve analysis of wet milled and dry milled procelain enamel' issued by the American Society for Testing and Materials (ASTM).

0.4 In reporting the result of a test made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard (Part I) prescribes methods for sieve analysis of wet-milled and dry-milled vitreous enamels.

2. TERMINOLOGY

2.1 For the purpose of this standard the definitions given in IS : 2717-1964† shall apply.

*Rules for rounding off numerical values (*revised*).

†Glossary of terms used in vitreous enamelware industry.

3. SAMPLING

3.0 Representative samples of vitreous enamels shall be drawn as follows depending on whether they have been wet-milled or dry-milled.

3.1 For Method A — Referee Method

3.1.1 *Wet-Milled Enamels* — Select a sample of about 100 g of slip representative of the material to be tested. Protect the samples from evaporation. Determine its water content by drying it to constant mass at a temperature not exceeding 122°C.

3.1.1.1 Stir and pass the slip through 425-micron IS Sieve, discarding the material retained on it, and weigh to the nearest 0.1 g a sample sufficient to contain 100 g of dry solids.

3.1.2 *Dry-Milled Enamels* — Select a sample representative of the material to be tested containing about 100 g of dry solids and weigh to the nearest 0.1 g.

3.2 For Method B — Routine Method — Select a sample of slip from the mill before unloading and pass it through 425-micron IS Sieve before weighing. Discard the material retained on the sieve. The sample shall consist of 100 g of slip for wet-milled enamel or 100 g for dry milled enamel.

4. METHOD A — REFEREE METHOD

4.0 General — Method A is intended for use where higher accuracy is required. In the case of a dispute, this method shall be used.

4.1 Apparatus

4.1.1 *Balance* — of at least 500 g capacity and accurate to 0.1 g.

4.1.2 *Sieves* — 425-micron, 75-micron and 45-micron IS Sieves (*see* IS : 460-1962*). The 45-micron sieve shall be used when the fineness is such that, from a sample containing 100 g of dry solids, less than 2 g is retained on 75-micron sieve.

NOTE 1 — It is recommended that the diameter and height of the sieves should be about 200 mm so that there is less possibility of flooding or splashing and they may fit properly in automatic tapping and shaking machines.

NOTE 2 — All sieves shall be standardized initially and after every 50 tests against a reference sieve by an organization authorized to do so. The correction for the sieve used in this test shall be determined by sieving tests made in accordance with **4.2**. Identical sample shall be sieved through the reference sieve and the test sieve. Test material shall be chosen so that 5 to 10 percent of it is retained on the reference sieve. The difference between the percentages of the residues on the reference and test sieves respectively is the amount of correction which shall be algebraically added to, or subtracted from the correction for the reference sieve to obtain the final correction. The 423-micron sieve need not be calibrated.

*Specification for test sieves *(revised)*.

IS : 8687 (Part I) - 1977

Indian Standard

METHODS OF TEST FOR VITREOUS ENAMELS AND FRITS

PART II FUSION FLOW TEST

UDC 666.293 : 539.571

INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

July 1978

Gr 2

AMENDMENT NO. 1 JUNE 2007
TO
IS 8687 (PART 2) : 1977 METHODS OF TEST FOR VITREOUS ENAMELS AND FRITS

PART 2 FUSION FLOW TEST

(*Page* 3, *clause* **2.1**, *line* 1) — Substitute 'IS 2717 1979†' *for* 'IS 2717 - 1964t†'.

(Page 3, *footnote marked* †) — Substitute the following for the existing

'†Glossary of terms relating to vitreous enamelware and ceramic metal systems (*first revision)'*

(CHD 9)

Reprography Unit, BIS, New Delhi, India

Indian Standard

METHODS OF TEST FOR VITREOUS ENAMELS AND FRITS

PART II FUSION FLOW TEST

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Indian Standard

METHODS OF TEST FOR VITREOUS ENAMELS AND FRITS

PART II FUSION FLOW TEST

0. FOREWORD

0.1 This Indian Standard (Part II) was adopted by the Indian Standards Institution on 10 October 1977, after the draft finalized by the Ceramicware Sectional Committee had been approved by the Chemical Division Council.

0.2 Determination of fluidity behaviour by fusion flow test is not carried out as an absolute method but as a comparative method. The test can be carried out quickly with simplified equipment to provide data about the fluidity behaviour of molten enamel. From the results of the flow test, conclusion on the processing ability of the enamel can be drawn in a more simplified manner than from the results of more expensive measurements with viscosity measuring instruments.

0.3 In the formulation of this standard assistance has been derived from Doc:N 31 E Determination of fluidity behaviour — Fusion flow test, issued by ISO/TC 107/SC 6 Vitreous and Porcelain Enamels, of the International Organization for Standardization.

0.4 In reporting the result of a test made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

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1. SCOPE

1.1 This standard (Part II) prescribes the method for determining the fluidity behaviour of vitreous enamels in viscous condition during firing.

NOTE — The fusion flow test may be used for molten enamels but not for sintered ground coat enamels.

2. TERMINOLOGY

2.1 For the purpose of this standard, the definitions given in IS: 2717-1964† shall apply.

*Rules for rounding off numerical values (*revised).*

†Glossary of terms used in vitreous enamelware industry.

3. PRINCIPLE

3.1 Enamels are ground dry or wet in accordance with processing conditions. Cylindrical specimens of specified mass are pressed from the test and the agreed reference enamels respectively which may be in the form of powder or dried slip.

3.2 The specimens are placed on an unglazed ceramic tile or matt ground-coat enamelled steel plate in horizontal position in the laboratory furnace at the temperature1 agreed upon, molten down to hemispherical shape and then permitted to flow with the tile or plate at an angle of 45° for an agreed period.

3.2.1 The flow number 'F_l' for length and the flow number 'F_b' for breadth are then calculated on the basis of the flow lengths and flow breadths of the specimens.

4. SAMPLING

4.1 A representative simple of the test and reference enamels shall be obtained, mixed thoroughly, and reduced by ??? to about 25 g.

5. APPARATUS

5.1 Reference Enamel - To be agreed upon between the parties concerned and to be similar in its flow characteristics to the enamel to be tested.

5.2 Ball Mill

5.3 Evaporating Device for example, hot-air oven, hot-plate, sand-bath, etc.

5.4 Mortar and Pestle

5.5 Balance accurate to 0.1 mg.

5.6 Press - for at least 5 MPa* pressure, and mould having an internal diameter of 8 mm, for the preparation of the specimens.

5.7 Flow Plate — a smooth prefired unglazed ceramic tile, square with a side length of 75 mm, thickness 5 to 6 mm and water absorption not exceeding 20 percent by mass at atmospheric pressure. A matt ground-coat-enamelled steel plate of dimensions mentioned above may also be used.

5.8 Tilting Frame - for placing the flow plate inside the laboratory furnace in horizontal position and subsequent tilting by 45° (*see* Fig. 1 and 2).

*1 MPa 10 kgf/cm^2 (approx).

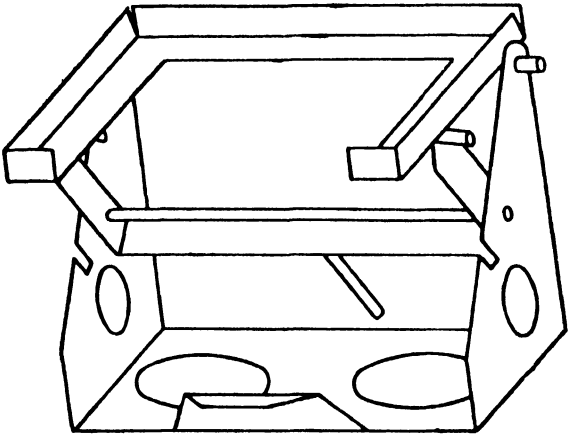
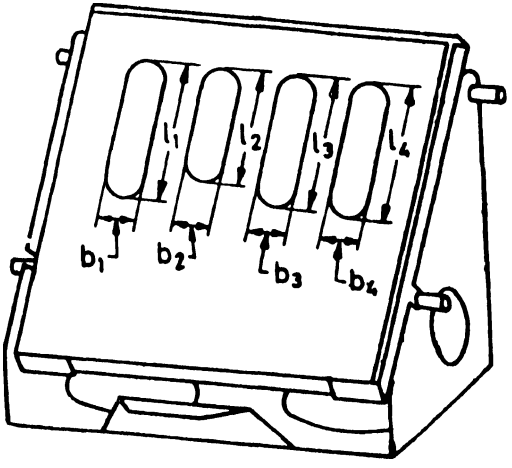


FIG.1 TILTING FRAME (SET FOR RECEIVING THE FLOW PLATE IN HORIZONTAL POSITION)



l_1, l_2, l_3 and l_4 are the flow lengths of the four specimens.

b_1, b_2, b_3 and b_4 are their maximal flow breadth.

FIG. 2 TILTING FRAME (SHOWN WITH FLOW PLATE AND FOUR SPECIMENS TILTED BY 45°)

5.9 Laboratory Furnace — electrically heated, allowing the temperature to be kept constant up to 900 ± 1°C.

5.10 Stop-Watch

6. TEST SPECIMENS

6.1 Preparation of Enamel — The sample may be taken from the ground enamel powder or may be ground separately in the ball mill. Mill additives and fineness of grinding depend on the conditions of processing.

NOTE 1 — If agreed, milling additives which are completely or partially soluble in water and are only used as a set-up agent, may be left out.

NOTE 2 — Where the fluidity of frits alone is to be tested, it should be milled dry.

6.1.1 Evaporate wet ground enamel to dryness in the evaporating dish. After cooling loosen the dried enamel and again transform into pulverized condition by means of the pestle and mortar.

6.2 Preparation of Test Specimens — Put one drop of water into the empty mould, and then 1 000 ± 10 mg of the enamel prepared according to **6.1** followed by another drop of water. Immediately thereafter, press the specimen at a pressure of at least 5 MPa, then take out and allow to dry for at least 30 minutes.

6.3 Number of Test Specimen — For each flow test, use one specimen made of the reference enamel and at least one specimen made of the enamel to be tested.

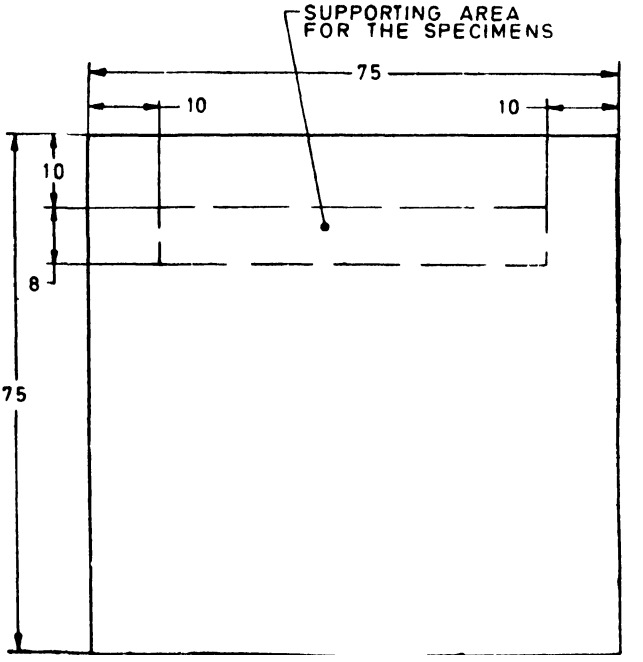
7. PROCEDURE

7.1 For each flow test place the requisite number of specimens of reference and test enamels (*see* **6.2**) on the flow plate at a distance of at least 8 mm within range of the supporting area (*see* Fig. 3).

7.2 Heat the laboratory furnace to the temperature agreed upon for respective enamels and carefully place the tilting frame, with flow plate (*see* **7.1**) in a horizontal position, into the furnace. When, the specimen made of reference enamel has become sufficiently soft to form approximately a hemisphere, tilt the flow plate by 45°.

NOTE — The time from placing the flow plate into the laboratory furnace until obtaining the hemispherical shape is to be determined by one or several pretests. This time is called the holding period.

7.3 At the end of the period agreed upon for the 45° position, the so-called flow period, take the flow plate out of the laboratory furnace.



All dimensions in millimetres.

FIG. 3 SUPPORTING AREA FOR SPECIMENS ON FLOW PLATE

7.4 Carry out at least two fusion flow tests for each determination with each set of specimens.

NOTE — During the various flow tests interchange the positions of the specimens made of enamel to be tested and of the specimen made of the reference enamel so that the influence of any temperature gradient which might be present in the furnace is eliminated.

8. EXPRESSION OF RESULTS

8.1 Measure the flow lengths and the maximal flow breadths of the reference and test enamels in millimetres (*see* Fig. 2) and calculate the flow number for each flow test as follows:

$$F_l = \frac{\text{Flow length of enamel to be tested}}{\text{Flow length of reference enamel}} \quad \ldots\ldots (1)$$

$$F_b = \frac{\text{Maximal flow breadth of enamel to be tested}}{\text{Maximal flow breadth of reference enamel}} \quad \ldots\ldots (2)$$

NOTE — If during a flow test several specimens of the enamel to be examined have been tested, the mean flow length and the mean maximal flow breadth of the specimens shall be used in equations (1) and (2).

9. TEST REPORT

9.1 The test report shall include the following particulars:

a) Designations of the reference and test enamels;

b) Temperature in the laboratory furnace;

c) Holding period of specimens in the laboratory furnace until formation of the hemisphere;

d) Flow period of specimens at 45° position of flow plate;

e) Number of specimens per fusion flow test;

f) Number of fusion flow test;

g) Flow number F_l and flow number F_b, single values and arithmetic means; and

h) Date of test.

INDIAN STANDARDS

ON

CERAMICWARE

IS:

2717-1964 Glossary of terms used in vitreous enamelwate industry
2781-1975 Glossary of terms relating to ceramicware *(first revision)*
2836-1974 Methods of test and quality requirements for porcelain laboratory apparatus *(first revision)*
2837 (Part I)-1975 Porcelain crucibles and basins : Part I Crucibles *(first revision)*
2837 (Part II)-1977 Porcelain a crucibles and basins : Part II Basins *(first revision*)
2858-1964 Sioneware containers for general purposes
2839-1964 Industrial stoneware
2840-1965 China clay for ceramic industry
2857-1976 Earthenware dinnerware (*first revision)*
3149-1968 Enamelware for home use (*first revision)*
3432-1965 Clay pipe triangles
3505-1965 Porcelain dinnerware
3936-1966 Poreclain mortars and pestles
3955-1966 High temperature ceramic combustion boats
3972-1968 Methods of test for viticous enamelware
3990-1967 High temperature ceramic combustion tubes
4589-1968 Ball clays for ceramic industry
5009-1968 Buchner funnels
5011-1968 Gooch crucibles
6154-1971 Perforated plates for desiccators
6988-1975 Fine china dinnerware
7087-1973 Ceramic tower packings
7402 (Part I)-1974 Ceramic water filters : Part I Filter containers
7402 (Part II)-1975 Ceramic water filters: Part II Filter candles
7775-1975 Ceramic grinding media and lining
8017-1976 Vitreous enamelled reflectors for use with ??? filament lamps
8687 (Part I)-1977 Methods of test for vitreous and frits : Part I Sieve analysis
8687 (Part II)-1977 Methods of test for vitreous and frits: Part II Fusion flow test

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